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Kinetics of release of carbon and carbon monoxide from a graphite furnace investigated by electrothermal vaporization inductively coupled plasma mass spectrometry

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Abstract—Detection of carbon-containing molecules released during the high temperature heating of a dry, unloaded HGA-type graphite furnace (GF) has been undertaken by interfacing the GF to an ICP-MS. Desorption of O_2 (as CO) chemisorbed onto the GF surface can be discriminated from that produced as a result of continuous oxidation of the surface by gaseous O_2 impurity. Because plasma-induced dissociation of CO is negligible, signals arising from C do not have to be deconvoluted from those of CO. To a first approximation, the formation of C and CO can be treated as independent processes. A theoretical analysis of the transient experimental signals is presented.

1. Introduction

A graphite furnace (GF), owing to its simplicity and versatility, is widely used as an atomizer in all versions of analytical atomic spectrometry including absorption [1], emission [2], fluorescence [3], and laser-enhanced spectrometry [4]. More recently, the GF has been frequently used as a vaporizer for ICP–MS [5]. Although introduced [1] into analytical spectrometry more than 30 years ago, only limited progress has been made in the understanding of the mechanisms of analyte atom formation in the GF. One of the reasons for this is the lack of reliable information dealing with the role of carbon in the atomization processes. It is presently well recognized that the graphite surface of the atomizer affects, to some extent, all aspects of analyte atom formation, either by direct interaction with the analyte or via its effect on the gas phase composition within the furnace. The most evident reaction is the formation of stable carbides of some analytes with the graphite of the furnace [6]. Surface reactivity plays an important role in the regulation of gas phase composition [7] and the surface concentration of active sites may determine the rates of analyte oxide reduction or carbidization [8, 9]. Numerous carbides have also been detected in the gas phase [10].

To understand the role of carbon in the operation of a GF a number of independent techniques have been applied. These include scanning electron microscopy [11], X-ray diffraction [12] and optical spectroscopy [13–16]. One of the most informative tools for this purpose is mass spectrometry, which permits simultaneous temporal detection of a number of species in the gas phase of the GF. The method, first introducted into

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